WADC TECHNICAL REPORT 53-16
PART 3

[redacted]

CORROSION PREVENTIVE ADDITIVES

E. J. SCHWOEGLER

L. U. BERMAN

ARMOUR RESEARCH FOUNDATION

ILLINOIS INSTITUTE OF TECHNOLOGY

DECEMBER 1954

WRIGHT AIR DEVELOPMENT CENTER

NOTICE

WADC TECHNICAL REPORT 53-16
PART 3

CORROSION PREVENTIVE ADDITIVES

E. J. SCHWOEGLER
L. U. BERMAN

ARMOUR RESEARCH FOUNDATION
ILLINOIS INSTITUTE OF TECHNOLOGY

DECEMBER 1954

MATERIALS LABORATORY
CONTRACT No. AF 33(038)-9202
PROJECT No. 3044
TASK No. 73311

WRIGHT AIR DEVELOPMENT CENTER
AIR RESEARCH AND DEVELOPMENT COMMAND
UNITED STATES AIR FORCE
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

Carpenter Litho & Prtg. Co., Springfield, O.
300 - 23 March 1955

FOREWORD

This report was prepared by the Armour Research Foundation under Contract No. AF 33(038)-9202. The contract was initiated under Project No. 3044, Aviation Lubricants, Task No. 73311, Corrosion Preventive Oils, formerly RDO No. 613-13, and was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Mr. R. W. Sneed acting as project engineer.

This report is the third in a series of reports on the same subject. The first report was published in February 1953 under the basic report number (WADC TR 53-16) only; it should be considered as Part 1 although it was not so marked. The second report, WADC TR 53-16 Pt 2, was issued in March 1954.

The purpose of this report is to evaluate the performance of a number of products for a specific application. Many of the materials tested were not developed or intended by the manufacturer for the conditions to which they have been subjected. Any failure or poor performance of a material is therefore not necessarily indicative of the utility of the material under less stringent conditions or for other applications.

ABSTRACT

This project was undertaken to study the development of new inhibitors that may replace or supplement petroleum sulfonates. The work was done by the Organic Chemistry Section of the Department of Chemistry and Chemical Engineering at Armour Research Foundation.

The evaluation of various effective inhibitors against corrosion of SAE 1020 steel in combination with sulfonates has shown that fair inhibition by one sulfonate, is improved by the addition of a small quantity of another inhibitor, usually an amine or amine salts. The amount added was less than that required for good inhibition by either compounds alone.

Over 200 compounds have been evaluated in the JAN-H-792 cabinet. Twelve of these passed the 100-hr requirement. Before using this test method, a study was made to determine reasons for lack of correlation between the results of the Armour Research Foundation cabinet and those of the Wright Air Development Center cabinets. Methods of correlating the cabinet conditions were found so that those compounds listed as passing would also pass the WADC JAN-H-792 test used in qualifying certain specification formulations. Six formulations containing barium dialkyl naphthalene sulfonate, phenothiazine and certain amine salts were also made which passed the 100-hr requirement.

The study of the mechanism of corrosion inhibition as related to structure and functional groups of the polar organic compounds was made on the data obtained from the galvanic system tests with a 52100 steel disk and cartridge brass clip. Most of the information was obtained from the tests conducted at 95% RH at 100°F over a 4-week period in the American Instrument Company cabinet. From the study in MIL-L-6085A base oil the sulfonates, certain amines, amine salts of carboxylic acids, certain oxidized petroleum fractions and certain fatty acid esters of pentaerythritol constituted the effective structures and groups. In the petroleum base oil, AN-O-6a, sulfonates of lower molecular weight, certain aliphatic amines, polyamine salts of carboxylic acids, certain N-alkyl- and aryl-substituted morpholine salts of carboxylic acids, glyoxalidines, and a few esters were effective.

Several additional compounds were synthesized from data obtained from previous test results but these did not show corrosion-inhibiting properties.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

M. R. WHITMORE
Technical Director
Materials Laboratory
Directorate of Research

TABLE OF CONTENTS

LIST OF TABLES

INTRODUCTION

This is a final report of work undertaken during the period 31 March 1953 to 31 March 1954 on a research project to develop and evaluate compounds as rust inhibitors which can replace or supplement petroleum sulfonates. Another objective of the project is to study the mechanism of corrosion inhibition based on the structural and functional groups of those polar organic compounds evaluated.

The major effort during the past year has been concerned with the evaluation and study of polar organic compounds as inhibitors in the JAN-H-792 humidity cabinet tests. This study was limited to the MIL-L-6085A base oil (MIL).

A study was also undertaken to determine the effects of combinations of various organic compounds as inhibitors for SAE 1020 steel in the Static Water Drop test.

Finally, the mechanism of corrosion inhibition as it concerned the structure of the polar compounds and the functional groups was studied. This study included the effects of various compounds in both AN-O-6a petroleum base oil (AN) and the synthetic diester oil, MIL-L-6085A (MIL), on the bimetallic system of 52100 steel-cartridge brass.

It also included a further study of the inhibitors found to be effective in the JAN-H-792 cabinet tests.

SECTION I

EVALUATION OF ORGANIC COMPOUNDS AS CORROSION INHIBITORS

In the course of this investigation, use was made of the Static Water Drop test, JAN-H-792 cabinet test, and a galvanic system test. For convenience, all solution concentrations given in this report are expressed as per cent by weight (wt %), unless otherwise specified.

1.1 Static Water Drop Test

The evaluation of organic compounds by the Static Water Drop test has been continued with the screening of a number of compounds at 0.2 % concentration in the two base oils. Table 1 lists the results obtained with these potential inhibitors. Pure fatty acids (No. 862 and 863) were found to be ineffective in both oils; these were used as a check on the commercial materials to insure that lack of potency was not due to inherent fatty acid impurities.

It may be significant that, in contrast to a 1-aminoethyl-2-alkenylglyoxalidine (No. 840) which showed no protective action when tested last year, the two 1-hydroxyethyl derivatives (No. 895 and 896) showed excellent inhibitory power, in spite of the limited solubility of one of them. These results are in accord with the observation, also obtained previously, with a commercial 1-hydroxyethyl derivative (No. 269). The "tailor-made" compounds (No. 897, 922, and 923) which were synthesized in an attempt to incorporate both corrosion- and oxidation-inhibiting properties into a single molecule unfortunately possess no corrosion-inhibiting properties in either base oil.

It had been observed previously (Final Report, 31 March 1952-31 March 1953), that in the case of certain amine salts of carboxylic acids, these products possess a potency well beyond that obtained with the original reactants alone. Thus, the Static Water Drop test has been employed, during the past period, for a study of the effects of combinations of inhibitors with the preparation of three series of two-component systems in MIL-L-6085A base oil. Three synthetic sulfonates, designated as "common components", were dissolved with each of 26 different compounds, chosen as group representatives of the various structures encountered. One compound (No. 776, sorbitan mono-oleate) previously showed no inhibiting properties (at 0.2 % concentration), in contrast to the other 25 which were excellent inhibitors. Each component in the combination was adjusted to 0.05 % concentration; in addition, each of the common components was run at 0.1 %, and, as a further check, the "group representatives" were tested alone at 0.05 % concentration. Table 2 presents a summary of the results obtained. From the data available at this point in the study, it appears that certain types of amines and amine derivatives may yield increased protective action in combination with these synthetic sulfonates.

1.2 Galvanic Test System

This test system, consisting of a 52100 ball-bearing steel disk coupled to a brass (Muntz metal) clip, was employed at 95 % static RH and 160 °F for a 3-week period in order to determine the effectiveness of organic compounds

Table 1

EVALUATION OF 19 COMPOUNDS
BY STATIC WATER DROP TESTS
(0.2 °/o conc)

Code No.	Compound	Corrosion Inhibition MIL*	Corrosion Inhibition AN*
862	Stearic acid (99+ °/o purity)	None	None
863	Linoleic acid (99+ °/o purity)	None	None
875	Pinonic acid salt of morpholine (1:1 mole ratio	Good	None
876	Pinonic acid salt of N-methylmorpholine (1:1 mole ratio)	None	
877	Elaidic acid (95 °/o purity)	None	None
879	N-(β-Ethoxyethyl) morpholine	None	Fair
880	Dioleoylamidoethylamine		None
881	Redistilled N-aminoethylmorpholine	Good	
882	Redistilled N-hydroxyethylmorpholine	Good	
883	N-(β-Diethylaminoethyl) morpholine	Good	Fair
887	Commercial corrosion inhibitor; low ash, S and P containing compound	None	None
888	N-(β-Dimethylaminoethyl)ethylenediamine	Good	Fair
889	1,2-Di-(N,N'-morpholino)ethane	Fair	None
895**	1-Hydroxyethyl-2-pentadecylglyoxalidine	Good	Good
896	1-Hydroxyethyl-2-undecylglyoxalidine	Good	Fair
897**	4,5-Dimorpholinocatechol	None	None
898	A sorbitan mono fatty acid ester	None	Fair
922	2,4,6-Tri-(morpholinomethyl)phenol	None	None
923**	Mannich reaction product of hydroquinone and morpholine	None	None

*MIL-L-6085, AN-O-6a.

**Saturated at 0.2 °/o conc and decanted fluid tested.

Table 2

EVALUATION OF COMBINATIONS OF INHIBITORS
IN MIL-L-6085A BASE OIL BY STATIC WATER DROP TEST

Code No.	Compound Type	Results with Alone	No. 352*	No. 351*	No. 116*
352	Sodium dialkyl naphthalene sulfonate*	Fair*			
351	Barium dialkyl naphthalene sulfonate*	Fair*			
116	Synthetic petroleum sulfonate*	Fair-good*			
109	2,4,6-Tri-(dimethylaminomethyl)-phenol	Good		Good	Good
162	N-Aminoethylmorpholine	Fair		Good	Good
176**	Phenylbiguanide	Good	Good	Good	Good
222	Morpholine	Good	Good	Good	Good
230	α-Methylbenzylmonoethanolamine	Fair	Good	Good	Good
246	Dinonylamine	No good	Good	Good	Good
248	t.-Octylamine	Good		Good	Good
255	Tributylamine	- - - -		Good	Good
269	1-Hydroxyethyl-2-heptadecenyl-glyoxalidine	No good		Good	Fair-good
293	Diphenylguanidine	Good		Good	Good
334**	Triethanolamine soap of tall oil	No good		Good	Good
466	Morpholine salt of 2-ethylhexoic acid, pH 6.7	No good		Good	Good
471	3,3'-Iminobispropylamine	Good		Good	Good
475	N-Hydroxyethylmorpholine	No good		Good	Good
477	2,5-Dimethylpiperazine	Good		Good	Good
490**	Hydrofuramide	No good		Good	Good

Table 2 (cont)

Code No.	Compound Type	Results with			
		Alone	No. 352*	No. 351*	No. 116*
493	N-(2-Hydroxyethyl)ethylenediamine	Good		Good	Good
583**	Morpholine salt of oleic acid, pH 9.0	No good		Good	Good
626	β-Phenylethylamine	Good		Good	Good
633	Morpholine salt of linoleic acid, pH 9.2	No good		Good	Fair-good
776	Sorbitan mono-oleate	No good		No good	No good
796**	Morpholine salt of sebacic acid (2:1 mole ratio), pH 6.0	Fair		Good	Good
803**	Morpholine salt of azelaic acid (2:1 mole ratio), pH 7.6	Good		Good	Good
820**	Morpholine salt of n-butylarsonic acid, pH 4.3	No good		Good	No good
846	Aminopropyl tallow amine	No good		Good	No good
851	N-(β-Dimethylaminoethyl)morpholine	Good		Good	Good

* At 0.1 % conc.

**Not completely soluble after 1 hr at 140°F; decanted fluid tested.

against corrosion in this type of bimetallic system. The details of the preparation of the specimens and the test procedure have been described previously.* Tables 3 and 4 list the results obtained with compounds in AN-O-6a and MIL-L-6085A base oils, respectively.

1.3 JAN-H-792 Humidity Cabinet Test

Included in most government specifications for corrosion-inhibiting formulations is a test which requires the compositions to be evaluated on 1010-1020 steel at 100% RH (with condensation) at 120°F. In order to qualify, the composition must prevent corrosion on the test panels during a 100-hr continuous exposure period; the details of this test are described in Spec JAN-H-792 and MIL-L-6085A.

Since WADC is the qualifying agency for Spec MIL-L-6085A formulations, it is essential that the results obtained in their JAN-H-792 test cabinets be reproduced in any other test cabinets. Results of preliminary tests in the cabinet at the Foundation (ARF) indicated that it was much too severe, by comparison with the results obtained by the qualifying agency (WADC).

First, a series of studies were initiated to determine the effects of polishing techniques. It was found that, although somewhat more time-consuming, the final polish with 310 emery paper yielded a test plate generally comparable to that polished with No. 600 SiC. It was further discovered that the quality of the plates chosen is of the utmost importance, since corrosion appears to be initiated and concentrated in the faint grooves, from the rolling marks, on the plates. It is, therefore, essential that the plates be chosen from selected prime sheet stock with the minimum amount of gouging and rolling marks and, further, that in the initial rough polish, the plates be ground or buffed well beyond these grooves, before the final polish is attempted.

Following the polishing studies, attention was focused on another important variable in the test: the flow rate of the air into the cabinet. Specification JAN-H-792 requires that the air flow be maintained between the following limits: 27.4-35.2 cu ft/hr at 70°F and 1-1/2 psi. The manometer supplied with the ARF cabinet was calibrated on a Precision Scientific Company wet test meter and extrapolated to 1-1/2 psi at 76°F, to obtain the following points: 35.2, 31.2, 27.4, 25.1, 20.5, and 17.7 cu ft/hr. The flow rate was maintained at a constant value by the use of two pressure regulators, in series. The initial experiments, made at 31.2 cu ft/hr, indicated that the ARF cabinet was operating too severely. So, a series of runs was made, under identical conditions except varying the air flow rate, with commercial formulations tested at WADC. Table 5, which lists the results of these tests, indicates that at 20.5 cu ft/hr at 76°F and 1-1/2 psi, the results obtained with the ARF cabinet are comparable to those of the WADC cabinets. The 76°F temperature, instead of 70°F, was chosen as more practical, since Spec JAN-H-792 requires that the cabinet be placed in a room maintained at 75°F ± 10° and Spec MIL-L-6085A requires that the plates be hung in a room maintained at 77°F ± 2°. Thus, the draining racks and the test cabinet may be located in close proximity. In addition, the air flow manometer would be calibrated at the operating equilibrium temperature to eliminate the need for

*WADC Technical Report 53-16, Parts 1 and 2.

Table 3

EVALUATION OF SEVEN ORGANIC COMPOUNDS
BY GALVANIC SPECIMEN TEST IN AN-O-6a BASE OIL
(0.2 °/₀ conc; 95 °/₀ RH; 160 °F)

Code No.	Compound	Results* on Disks
478	Composition restricted	Faint stain under central hump; remainder of surface, good.
479	Reaction product of lauric acid and diethylenetriamine	Stain and one tiny pit under central hump; remainder of surface, good.
481	Reaction product of oleic acid and diethylenetriamine	Stain and a few pits under central hump.
482	Reaction product of lauric acid and N-(2-hydroxyethyl)-ethylenediamine	Stain under central hump of clip and one pit at close contact without aeration; remainder of surface, good.
491	Triethylenetetramine	Stain under central hump and one tiny pit at close contact without aeration.
503	Morpholine salt of 2-ethyl-hexoic acid (10 °/₀ mole excess acid) pH 6.0	Light stain under central hump.
504	Morpholine salt of 2-ethyl-hexoic acid (10 °/₀ mole excess amine) pH 7.7	Stain and two tiny pits under central hump and one tiny pit at close contact without aeration.

*All clips were satisfactory.

Table 4

EVALUATION OF 21 ORGANIC COMPOUNDS
BY GALVANIC SPECIMEN TEST IN MIL-L-6085A BASE OIL
(0.2 °/₀ conc; 95 °/₀ RH; 160 °F)

Code No.	Compound	Results	
		Clips	Disks
174	1,3-Diethylthiourea	Stained green at contact area	Numerous tiny pits at close contact without aeration.
175	1,3-Diisopropylthiourea	OK	Faint stain and a few tiny pits under central hump; numerous tiny pits surrounding central hump at close contact without aeration.
178	Sodium 2-ethylhexylsulfate	Stained	Faint stain under central hump.
200	Alkylene oxide condensation products	Stained	Light stain under central hump.
201	Alkylene oxide condensation products	Stained	Mottled stain under central hump.
205	Alkylene oxide condensation products	OK	Mottled stain (and a pit in one disk) under central hump.
206	Alkylene oxide condensation products	OK	Mottled stain and a large pit under central hump; remainder of surface, good.
209	Alkylene oxide condensation products	Stained under central hump	Mottled stain and two pits under central hump; remainder of surface, good.
214	Alkylene oxide condensation products	OK	Green stain under central hump and one pit at close contact without aeration; remainder of surface, good.
216	Alkylene oxide condensation products	OK	Mottled stain under central hump; remainder of surface, good.
322	Sulfonated petroleum products, butyl alcohol, and selected mineral oils	Stained green	NO CORROSION under clip area; remainder of surface, excellent.
348	Synthetic petroleum sulfonate	OK	NO CORROSION under clip area; remainder of suface, good.
466	Morpholine salt of 2-ethylhexoic acid, pH 6.7	OK	Very, very faint stain under central hump; remainder of surface, good.

Table 4 (cont)

Code No.	Compound	Results	
		Clips	Disks
479	Reaction product of lauric acid and diethylenetriamine	OK	Stain (and two pits in one disk) under central hump with one tiny pit (in one disk) at close contact without aeration; remainder of surface, good.
481	Reaction product of oleic acid and diethylenetriamine	OK	Stain under central hump and one tiny pit at close contact without aeration; remainder of surface, good.
490	Hydrofuramide	OK	Stain (and one tiny pit in one disk) under central hump; remainder of surface, good.
493	N-(2-Hydroxyethyl)-ethylenediamine	One stained under central hump	Stain with one pit under central hump and one pit at close contact without aeration.
497	Composite of partially sulfonated waxylbenzenesulfonate Fractions 3, 4, and 5	OK	Very faint stain under central hump; remainder of surface, good.
499	Waxylbenzenesulfonate, Fraction 4	Clips lightly stained at contact areas	Very faint stain under central hump; remainder of surface, good.
503	Morpholine salt of 2-ethylhexoic acid (10% mole excess acid) pH 6.0	OK	Light mottled stain under central hump; remainder of surface, good.
504	Morpholine salt of 2-ethylhexoic acid (10% mole excess amine) pH 7.7	Light mottled stain on contact areas	Faint stain under central hump; remainder of surface, good.

double calibration or interpolation of calibrations between operating temperature and 70 °F.

Table 5

EFFECT OF AIR FLOW RATE ON COMMERCIAL MIL-L-6085A FORMULATIONS IN JAN-H-792 CABINET AFTER 100-HOUR EXPOSURE (Duplicate test plates)

	Corrosion			
Code No.	31.2*	27.4*	25.1*	20.5*
878	Severe	Few 2-mm spots	Few > 1-mm spots	None
884		Few 2-5-mm spots	Few 2-4-mm spots	None
885	Severe	Numerous > 1-mm spots	Several > 1-mm spots	Few >1-mm spots
886**		Severe at 24-hr	Severe at 24-hr	Severe
892		Numerous > 1-mm spots	Numerous > 1-mm spots	None
894				1 >1-mm pit

*Cu ft air/hr at 76°F, 1-1/2 psi.

**Failed at WADC.

The specimen for the JAN-H-792 cabinet test is a cold-rolled (or drawn) panel, 2 x 4 x 1/8 in., made of 1018 steel conforming to Spec QQ-S-636. Drawing or rolling marks are invariably present on the surface and sometimes penetrate to a considerable depth below the surface. If prime selected stock is obtained, these crevices are usually shallow, so that the initial grinding and polishing operation is relatively easy. Some plates were sent to a commercial shop where they were ground well beyond the rolling marks left by a No. 240 SiC belt grinder. (With badly gouged plates, a No. 150 or even a No. 50 SiC belt was required for the first rough grind.) Subsequently, the plates were polished with a No. 320 SiC belt, then buffed to a high polish, immediately coated with uninhibited mineral oil, and finally wrapped in tissue paper. Forceps are employed for the handling of the plates in the laboratory procedure.

1. The plates are swept through carbon tetrachloride to degrease them and were finally wiped with a clean towel to remove any nongrease film.

2. The plates are then swept through 50 °/ₒ methanol, just below the boiling point, again wiped with a clean towel, and immediately stored in dry CP benzene.

3. Each plate, secured by means of a wooden jig, is polished with 3/0 emery paper (3-in. width) to a uniform satin finish on both sides, on a rubber-backed 12-in. diameter wheel,* revolving at 36-38 rpm.

4. After polishing, the plates are stacked lengthwise, in fresh CP benzene; care is taken not to scratch or gouge the critical area of the polished surface.

---

* Q.A.W. Wheel, manufactured by Minnesota Mining and Manufacturing Company.

5. The plates are given a final degreasing, identical with the Static Water Drop test plates* and stored in a desiccator over Drierite for a period not exceeding 24 hr.

6. The plates are then treated (dipped, drained, etc) as required by Spec MIL-L-6085A and exposed in the JAN-H-792 cabinet operating at an air flow rate of 20.5 cu ft/min (at 76 °F and 1-1/2 psi).

In this test a panel is said to fail when either of the following conditions is observed in the significant panel area (defined in the specification), after 100 hr of exposure:

1. A corroded area of 2-mm diameter or larger
2. Two or more spots of between 1- and 2-mm diameter.

Table 6 lists the results of the test following the procedure described on a variety of organic compounds at 5 % concentration in MIL-L-6085A base oil. All of the compounds tested had shown good or fair inhibiting properties previously in the Static Water Drop test. In addition, several synthetic blends (Code 899-914 inclusive), prepared in this laboratory, were also evaluated in this test. Table 6 gives, generally, the time of corrosion over the total significant area in hours. Also, in cases of failure just at, or before, 100-hr exposure, the degree of failure is noted. In certain cases, according to the specification, the compounds can be considered to have passed the test (numerous $<$1-mm spots), but a differentiation between this condition and that of complete absence of corrosion was considered noteworthy.

SECTION II

SYNTHESIS OF POSSIBLE NEW CORROSION INHIBITORS

During the past contract period, several additional compounds have been synthesized to assist in the development of new corrosion inhibitors. These preparations were synthesized on the basis of results of evaluation of various organic compounds with certain structural and functional group characteristics.

Any compound which possesses the requisite corrosion-inhibiting properties for use in Spec MIL-L-6085A must be combined with a suitable oxidation inhibitor before an acceptable formulation can be blended. It would be very desirable to prepare a single compound having both the properties of an oxidation and corrosion inhibitor. Therefore, attempts were made to synthesize such compounds.

Morpholine, which has shown excellent corrosion inhibition by the Static Water Drop test, was condensed with catechol (an oxidation inhibitor) according to published directions** to obtain 4,5-dimorpholinocatechol. Unfortunately, this compound, which was extremely insoluble in both base oils, showed no inhibiting properties (Table 1).

---

* WADC Technical Report 53-16, Parts 1 and 2.

**Henry and Dehn. J. Am. Chem. Soc. 74, 278, 1952.

Table 6

EVALUATION OF 239 POSSIBLE INHIBITORS IN MIL-L-6085A BASE OIL BY 100-HOUR JAN-H-792 CABINET TESTS

Code No.	Compound	Time of Corrosion, hr
101*	Strontium petroleum sulfonate	NO CORROSION at 100
104*	Dimethylaminomethylphenol	100
109	2,4,6-Tri-(dimethylaminomethyl)phenol	24
116	Synthetic petroleum sulfonate	24-72
118*	Amido alcohol	24
121	t.-Alkylamine of 12-15 C atoms	24
125*	Pentaerythritol mono-oleate	24-48
130*	Pentaerythritol monostearate	48-72
131*	Petroleum sodium sulfonate	Several 2-6-mm spots at 100
142	Organic acids from oxidized petroleum fraction (MW = 450-490)	24
144*	Oxidized petroleum fraction containing lead soaps of acid	90
162	N-Aminoethylmorpholine	24
176*	Phenylbiguanide	24
185*	Sodium petroleum sulfonate	24
190*	Phosphoric acid-amine condensation product	24-48
194*	Tetraethylenepentamine	24
196*	Hydrocarbon oil oxidation products	96
222	Morpholine	24
226	Mixture of oleic and linoleic acids	24-48
230	α-Methylbenzylmonoethanolamine	24
231	α-Methylbenzylamine	100
237	Composition restricted	24
239*	A sulfonated, high-molecular-weight base	Numerous 4-6-mm spots at 100
246	Dinonylamine	24
247	Benzyldimethylamine	24
248	t.-Octylamine	100
255	Tributylamine	100
269*	1-Hydroxyethyl-2-heptadecenylglyoxalidine	24

Table 6 (cont)

Code No.	Compound	Time of Corrosion, hr
293*	Diphenylguanidine	24
308*	Reaction products of ethyl chloride, formaldehyde, and ammonia	24
317*	Reaction product of acetaldehyde and aniline	24-48
322	Sulfonated petroleum products, butyl alcohol and selected mineral oils	NO CORROSION at 100
330*	Dibutylammonium oleate	24-48
334*	Triethanolamine soap of tall oil	24
346*	Oxidized petroleum fraction, containing lead and sodium salts of acids present	72
348	Synthetic petroleum sulfonate	100
349	High-molecular-weight sodium petroleum sulfonate	100
351	Barium dialkyl napthalene sulfonate	NO CORROSION at 100
352	Sodium dialkyl naphthalene sulfonate	Numerous 2-6-mm spots at 100
357*	Substituted glyoxalidine	24-48
358*	Substituted glyoxalidine	24-48
465*	N-Aminoethylmorpholine salt of 2-ethylhexoic acid, pH 7.3	24
466	Morpholine salt of 2-ethylhexoic acid, pH 6.7	24
471*	3,3'-Iminobispropylamine	48-96
473	N-Aminopropylmorpholine	24
475	N-Hydroxyethylmorpholine	24
477*	2,5-Dimethylpiperazine	24
478*	Composition restricted	96
482*	Reaction product of lauric acid and aminoethylethanolamine	24
490*	Hydrofuramide	16
491*	Triethylenetetramine	48-96
493*	N-(2-Hydroxyethyl)ethylenediamine	48-96
497*	Composite of waxylbenzene sulfonate Fractions 3,4, and 5; partially sulfonated	96

Table 6 (cont)

Code No.	Compound	Time of Corrosion, hr
499*	Waxylbenzene sulfonate, Fraction 4	Several 6-mm spots at 100
501	Triethylamine	24
503	Morpholine salt of 2-ethylhexoic acid (10 % mole excess acid) pH 6.0	24
504	Morpholine salt of 2-ethylhexoic acid (10 % mole excess amine) pH 7.7	24
505*	N-Aminoethylmorpholine salt of 2-ethylhexoic acid (10 % mole excess acid) pH 6.5	24
506*	N-Aminoethylmorpholine salt of 2-ethylhexoic acid (10 % mole excess amine) pH 8.1	24
507	N-Methylmorpholine salt of 2-ethylhexoic acid (10 % mole excess acid) pH 5.8	24
508	N-Methylmorpholine salt of 2-ethylhexoic acid (10 % mole excess amine) pH 6.6	24
509	N-Aminopropylmorpholine salt of 2-ethylhexoic acid (10 % mole excess acid) pH 7.4	24
510	N-Aminopropylmorpholine salt of 2-ethylhexoic acid (10 % mole excess amine) pH 8.0	24
511*	N-Hydroxyethylmorpholine salt of 2-ethylhexoic acid, pH 6.0	24
523*	Petroleum sulfonate	24-48
542	t.-Octylamine salt of 2-ethylhexoic acid, pH 6.5	24
543	Tributylamine salt of 2-ethylhexoic acid, pH 6.9	24
544*	Benzyldimethylamine salt of 2-ethylhexoic acid, pH 6.3	24
546	α-Methylbenzylamine salt of 2-ethylhexoic acid, pH 6.6	24
547*	α-Methylbenzyldiethanolamine salt of 2-ethylhexoic acid, pH 6.6	24
563	Morpholine salt of 2-ethylhexoic acid**	16
564*	N-Aminoethylmorpholine salt of 2-ethylhexoic acid**	24

Table 6 (cont)

Code No.	Compound	Time of Corrosion, hr
565*	N-Methylmorpholine salt of 2-ethylhexoic acid**	24
566	N-Aminopropylmorpholine salt of 2-ethylhexoic acid**	24
567*	N-Hydroxyethylmorpholine salt of 2-ethylhexoic acid**	24
569*	3, 3'-Iminobispropylamine salt of 2-ethylhexoic acid**	24
570	α-Methylbenzylmonoethanolamine salt of 2-ethylhexoic acid**	24
571	Dinonylamine salt of 2-ethylhexoic acid**	24
572	t.-Octylamine salt of 2-ethylhexoic acid**	24
573	Tributylamine salt of 2-ethylhexoic acid**	24
574*	Benzyldimethylamine salt of 2-ethylhexoic acid**	24
576*	α-Methylbenzylamine salt of 2-ethylhexoic acid**	24
577	α-Methylbenzyldiethanolamine salt of 2-ethylhexoic acid**	24
582	α-Methylbenzylmonoethanolamine salt of 2-ethylhexoic acid, pH 7.5	24
583	Morpholine salt of oleic acid, pH 9.0	72
584	Dinonylamine salt of oleic acid, pH 10.1	24-48
585*	N-Aminoethylmorpholine salt of oleic acid, pH 8.8	NO CORROSION at 100
587	N-Methylmorpholine salt of oleic acid, pH 8.2	Mild at 100
588*	N-Aminopropylmorpholine salt of oleic acid, pH 9.3	Numerous < 1-mm spots at 100
592	t.-Octylamine salt of oleic acid, pH 9.5	Mild at 100
594*	Benzyldimethylamine salt of oleic acid, pH 8.3	Mild at 100
595*	Tetraethylenepentamine salt of oleic acid, pH 10.3	NO CORROSION at 100
596	α-Methylbenzylamine salt of oleic acid, pH 8.6	NO CORROSION at 100
600	α-Methylbenzylmonoethanolamine salt of oleic acid, pH 8.4	NO CORROSION at 100

Table 6 (cont)

Code No.	Compound	Time of Corrosion, hr
601*	α-Methylbenzyldiethanolamine salt of oleic acid, pH 7.9	48
602*	Morpholine salt of oleic acid**	72
603	N-Aminoethylmorpholine salt of oleic acid**	48-96
604*	3, 3'-Iminobispropylamine salt of oleic acid**	96
605	N-Methylmorpholine salt of oleic acid**	48
606	N-Aminopropylmorpholine salt of oleic acid**	NO CORROSION at 100
610	Dinonylamine salt of oleic acid**	24
611	t.-Octylamine salt of oleic acid**	NO CORROSION at 100
613	Benzyldimethylamine salt of oleic acid**	16
614*	Tetraethylenepentamine salt of oleic acid**	NO CORROSION at 100
615	α-Methylbenzylamine salt of oleic acid**	16
619	α-Methylbenzylmonoethanolamine salt of oleic acid**	Numerous spots <1-mm at 72
620*	α-Methylbenzyldiethanolamine salt of oleic acid**	48
626	β-Phenylethylamine	24
633*	Morpholine salt of linoleic acid, pH 9.2	24
635*	3, 3'-Iminobispropylamine salt of linoleic acid, pH 11.2	Several 2-6-mm spots at 100
636	N-Methylmorpholine salt of linoleic acid 8.3	24-48
642	t.-Octylamine salt of linoleic acid, pH 9.8	96
643	Tributylamine salt of linoleic acid, pH 8.0	24-48
644	Benzyldimethylamine salt of linoleic acid, pH 8.7	24-48
645*	Tetraethylenepentamine salt of linoleic acid, pH 10.5	Several 4-6-mm spots at 100
646	α-Methylbenzylamine salt of linoleic acid, pH 8.5	24-48
649*	3, 3'-Iminobispropylamine salt of linoleic acid**	NO CORROSION at 100

Table 6 (cont)

Code No.	Compound	Time of Corrosion, hr
650*	N-Methylmorpholine salt of linoleic acid**	96
651	N-Aminopropylmorpholine salt of linoleic acid**	NO CORROSION at 100
652*	N-Hydroxyethylmorpholine salt of linoleic acid**	96
656	t.-Octylamine salt of linoleic acid**	96
657	Tributylamine salt of linoleic acid**	96
658	Benzyldimethylamine salt of linoleic acid**	24-48
659*	Tetraethylenepentamine salt of linoleic acid**	96
660	α-Methylbenzylamine salt of linoleic acid**	24-48
661*	Morpholine salt of pelargonic acid, pH 7.3	24
662*	N-Aminoethylmorpholine salt of pelargonic acid, pH 7.6	24-48
663*	3, 3'-Iminobispropylamine salt of pelargonic acid, pH 10.3	24-48
664	N-Methylmorpholine salt of pelargonic acid, pH 7.0	72
665	N-Aminopropylmorpholine salt of pelargonic acid, pH 8.0	24-48
666*	N-Hydroxyethylmorpholine salt of pelargonic acid, pH 6.8	96
669	Dinonylamine salt of pelargonic acid, pH 10.2	24
671	Tributylamine salt of pelargonic acid, pH 7.6	72
672	Benzyldimethylamine salt of pelargonic acid, pH 7.1	96
673*	Tetraethylenepentamine salt of pelargonic acid, pH 9.7	16
674	α-Methylbenzylamine salt of pelargonic acid, pH 7.5	96
677	Octylphenoxyethoxyethyldimethylamine salt of pelargonic acid, pH 6.1	96

Table 6 (cont)

Code No.	Compound	Time of Corrosion, hr
678	α-Methylbenzylmonoethanolamine salt of pelargonic acid, pH 7.3	48
679*	α-Methylbenzyldiethanolamine salt of pelargonic acid, pH 7.0	48
680*	Morpholine salt of pelargonic acid**	16
681*	N-Aminoethylmorpholine salt of pelargonic acid**	96
682*	3,3'-Iminobispropylamine salt of pelargonic acid**	96
683	N-Methylmorpholine salt of pelargonic acid**	16
684*	N-Aminopropylmorpholine salt of pelargonic acid**	96
685*	N-Hydroxyethylmorpholine salt of pelargonic acid**	96
689*	t.-Octylamine salt of pelargonic acid**	24
690	Tributylamine salt of pelargonic acid**	72
691*	Benzyldimethylamine salt of pelargonic acid**	24
692*	Tetraethylenepentamine salt of pelargonic acid**	24
693	α-Methylbenzylamine salt of pelargonic acid**	24-48
695	N-(β-Hydroxypropyl)-o-toluidine salt of pelargonic acid**	72
696	Octylphenoxyethoxyethyldimethylamine salt of pelargonic acid**	72
697*	α-Methylbenzylmonoethanolamine salt of pelargonic acid**	72
698*	α-Methylbenzyldiethanolamine salt of pelargonic acid**	72
701*	3,3'-Iminobispropylamine salt of nicotinic acid, pH 10.3	48
711*	Tetraethylenepentamine salt of nicotinic acid, pH 9.5	48
720*	3,3'-Iminobispropylamine salt of nicotinic acid**	48
730*	Tetraethylenepentamine salt of nicotinic acid**	48

Table 6 (cont)

Code No.	Compound	Time of Corrosion
738	Ethylenediamine	72
739	Propylenediamine	24
742*	Commerical anionic surfactant	24
744*	Cyclohexylamine salt of commercial anionic surfactant	72-96
747*	Octylphenoxyethoxyethyldimethylamine salt of linoleic acid, pH 7.5	Starting by 100 (numerous $<$1-mm spots)
748	α-Methylbenzylmonoethanolamine salt of linoleic acid, pH 8.4	72
749*	α-Methylbenzyldiethanolamine salt of linoleic acid, pH 8.0	72
753	Di-2-ethylhexylamine salt of oleic acid, pH 8.5	72
757	Di-2-ethylhexylamine salt of pelargonic acid, pH 6.2	72
764	α-Methylbenzylmonoethanolamine salt of linoleic acid**	72
765*	α-Methylbenzyldiethanolamine salt of linoleic acid**	24-48
773	Di-2-ethylhexylamine salt of linoleic acid**	24
774	Di-2-ethylhexylamine salt of pelargonic acid**	24
776	Sorbitan mono-oleate	48
783*	Barium sulfonate (MW = 991)	48-72
784*	Calcium sulfonate (MW = 884)	96
785	Strontium sulfonate (MW = 942)	72
790*	N-Aminoethylmorpholine salt of adipic acid (2:1 mole ratio) pH 5.4	24
791*	3,3'-Iminobispropylamine salt of adipic acid (2:1 mole ratio) pH 9.8	24
792*	N-Hydroxyethylmorpholine salt of adipic acid (2:1 mole ratio) pH 5.3	24
793*	t.-Octylamine salt of adipic acid (2:1 mole ratio) pH 4.9	24
794*	Tetraethylenepentamine salt of adipic acid (2:1 mole ratio) pH 9.3	24
796*	Morpholine salt of sebacic acid (2:1 mole ratio) pH 6.0	24

Table 6 (cont)

Code No.	Compound	Time of Corrosion, hr
797*	N-Aminoethylmorpholine salt of sebacic acid (2:1 mole ratio) pH 7.7	24
798*	3,3'-Iminobispropylamine salt of sebacic acid (2:1 mole ratio) pH 9.9	24
799*	N-Hydroxyethylmorpholine salt of sebacic acid (2:1 mole ratio) pH 6.0	24
800*	t.-Octylamine salt of sebacic acid (2:1 mole ratio) pH 6.2	24
801*	Tetraethylenepentamine salt of sebacic acid (2:1 mole ratio) pH 9.6	24
805*	3,3'-Iminobispropylamine salt of azelaic acid (2:1 mole ratio) pH 10.5	48
806*	N-Hydroxyethylmorpholine salt of azelaic acid (2:1 mole ratio) pH 6.6	48
807*	t.-Octylamine salt of azelaic acid (2:1 mole ratio) pH 8.9	48
810*	3,3'-Iminobispropylamine salt of 2-ethylhexoic acid, pH 10.1	48
812*	n-Butylarsonic acid	48
818	3,3'-Iminobispropylamine salt of mahogany sulfonic acid, pH 9.9	NO CORROSION at 100
819*	Tetraethylenepentamine salt of mahogany sulfonic acid, pH 9.5	72
823*	t.-Octylamine salt of n-butylarsonic acid, pH 4.3	48
826	N-(2-Hydroxypropyl) morpholine	24
827	N-(3-Hydroxypropyl) morpholine	24
843*	Kerosene amines	24
845	Aminopropyl soya amine	Numerous 2-6-mm spots at 24
846*	Aminopropyl tallow amine	Numerous 2-6-mm spots at 24
847	Aminopropyl lauric amine	Numerous 2-6-mm spots at 24
848	Aminopropyl cocoanut amine	Numerous 2-6-mm spots at 24
849	2-Amino-n-octane	24
851	N-(β-Dimethylaminoethyl) morpholine	72

Table 6 (cont)

Code No.	Compound	Time of Corrosion
852	Monoethylamine	72
853	Diethylamine	72
854	Dibutylamine	72
856	Di-iso-propylamine	72
857	n-Heptylamine	24
858	n-Octylamine	24
860	n-Decylamine	24
864	n-Propylamine	24
865	Dipropylamine	24
866	Tripropylamine	24
867	sec.-Butylamine	24
868	iso-Butylamine	24
869	Di-iso-butylamine	24
870	2-Aminopentane	24
874	N-(4-Hydroxybutyl)morpholine	24
881	Distillate from commercial N-hydroxyethyl-morpholine	24
883	N-(β-Diethylaminoethyl)morpholine	24
888	N-(β-Dimethylaminoethyl)ethylenediamine	24
889	1,2-Di(N,N'-morpholino)ethane	24
897	4,5-Dimorpholino catechol	24
898	A sorbitan mono fatty acid ester	48
***		
899	ARF formulation	A few 2-mm spots at 24
900	ARF formulation	A few 2-mm spots at 24
901	ARF formulation	A few 4-6-mm spots at 48
902	ARF formulation	Several 4-6-mm spots at 72
903	ARF formulation	Two 2-4-mm spots at 72
904	ARF formulation	two 2-mm spots at 48
905	ARF formulation	Several 2-4-mm spots at 100
906	ARF formulation	Three 4-6-mm spots at 100

Table 6 (cont)

Code No.	Compound	Time of Corrosion, hr
907	ARF formulation	NO CORROSION at 100
908	ARF formulation	NO CORROSION at 100
909	ARF formulation	NO CORROSION at 100
910	ARF formulation	NO CORROSION at 100
911	ARF formulation	Several $<$1-mm spots at 100
912	ARF formulation	NO CORROSION at 100
914	ARF formulation	NO CORROSION at 100

*Saturated at 5 %. conc.

**Heated 3 hr at 150 °C.

***See compositions in APPENDIX

Next, cognizance was taken of the fact that the Static Water Drop test indicated 2,4,6-tri-(dimethylaminomethyl)phenol was an excellent inhibitor in MIL-L-6085A base oil and fair in AN-O-6a. A Mannich reaction was executed on phenol and morpholine, as described in the literature,* to obtain 2,4,6-tri-(morpholinomethyl)phenol. In addition, the Mannich reaction between morpholine and hydroquinone was carried out in a manner comparable to that for a similar compound.** Although analytical data was not available for its proof of structure, the reaction product obtained can be presumed to be 2,5-di-(morpholinomethyl)hydroquinone. Both compounds were inactive corrosion inhibitors in either base oil (Table 1).

Attempts to condense N-aminoethylmorpholine with aminoethyl bromide hydrobromide, under mild alkaline conditions, yielded no significant amount of reaction product. This condensation was attempted in the hope of obtaining a polyethylamine, substituted with morpholine on one end. Such a compound could be employed in the synthesis of a 1,2-disubstituted glyoxalidine, with a morpholinoethyl, or (morpholinoethyl)polyaminoethyl group in the 2-position. A compound of this type, containing two heterocyclic nuclei, should possess some very unusual properties.

SECTION III

MECHANISM OF CORROSION INHIBITION

In this project the mechanism of corrosion inhibition was studied by investigating the effect of the structure of polar organic compounds and of the functional groups on inhibition of corrosion on SAE 1020 steel and on the galvanic system, cartridge brass-52100 steel. These were evaluated by the Static Water Drop test and a galvanic test at definite relative humidity and temperature conditions.

In addition, three series of compounds were evaluated in different pairs in order to supply additional information concerning the effect of association. These pairs were evaluated by the Static Water Drop test on SAE 1020 steel.

3.1 Inhibition in a Galvanic System

The mechanism of corrosion inhibition in the bimetallic system was studied with a 52100 steel disk (ball-bearing steel) with an attached cartridge brass clip. Three sets of relative humidity-temperature conditions were used. One was at 50 % RH at 77 °F by the method described in MIL-L-6085A. A second was at 95 % RH at 100 °F. The third was similar to that described in MIL-L-6085A, except that it was conducted at 95 % RH at 160 °F.

These studies were carried out on both the hydrocarbon base oil (AN-O-6a) and the synthetic diester base oil (MIL-L-6085A) with 0.2 % concentrations of organic compounds.

---

*Bruson and MacMullen. J. Am. Chem. Soc. 63, 270, 1941.

**Caldwell and Thompson, ibid, 61, 765, 1939.

3.1.1 50 °/₀ Relative Humidity at 77°F

Table 7 gives the results of tests on 15 compounds carried out in the hydrocarbon oil for a 10-day period. Two of the three sulfonates evaluated were effective in AN-O-6a base oil. Compound 116 is supposedly prepared from a petroleum stock and No. 352 is a synthetic dialkyl naphthalene sulfonate. The alkyl groups are said to be nonyl groups obtained by using propylene trimer as the alkylating agent. The strontium salt of a petroleum sulfonic acid (No. 101) was ineffective; the sulfonic acid was not similar to that used to prepare No. 116.

Of the seven amines evaluated, the polyamino compounds were ineffective. It also appears that amine effectiveness is not a function of molecular weight.

Two glyoxalidines of widely differing molecular weights were effective.

Results from a similar study with the diester oil are given in Table 8. These results are even less conclusive from the mechanism standpoint since all except one compound were effective. Although the amines differed both in structure and in molecular weight, all of them were effective. The only ineffective material was 1-hydroxyethyl-2-heptadecenylglyoxalidine.

3.1.2 95 °/₀ Relative Humidity at 100°F

The first series in this study was carried out for a 19-day period in the American Instrument Company humidity cabinet. A second series involving the evaluation of about 60 compounds in the AN-O-6a base oil and nearly 100 in the MIL-L-6085A base oil was carried out over a 4-week test period.

With the 19-day test period a total of 27 compounds were evaluated in the two oils. The results in the AN-O-6a base oil are summarized in Table 9. All the oxidized petroleum fractions and derivatives studies were effective, except those which contained only the lead soaps of the acids present. Even those containing lead soaps were effective if there were some sodium salts present with the lead soaps. The two sulfonates which were effective are in the same molecular weight range. Morpholine was effective; that N-aminoethylmorpholine was not may be due to the presence of the primary amino group in it.

Similarly, in the MIL-L-6085A base oil (Table 10) only a few compounds were evaluated and most of these were effective. Dimethylaminomethylphenol was effective, whereas the trisubstituted 2,4,6-tri-(dimethylaminomethyl)-phenol was not. The ineffectiveness of the latter compound could be due to some effect of the phenol group or to the difference in symmetry from that of the first compound; this would allow for a less effective adsorption. An insufficient number of compounds were evaluated in this test to permit generalizations for either oil.

Over the four-week period a larger number of compounds were evaluated, and, since this longer period gave a more selective screening, the results have more significance. Only 30 °/₀ of those compounds evaluated in

Table 7

EVALUATION OF 15 COMPOUNDS BY GALVANIC TEST*
IN AN-O-6a BASE OIL

Code No.	Compound	Result
	Sulfonates	
116	Synthetic sodium petroleum sulfonate	Good
352	Sodium dialkyl naphthalene sulfonate	Good
101	Strontium petroleum sulfonate	Ineffective
	Amines**	
222	Morpholine (86)	Good
248	t.-Octylamine (129)	Good
501	Triethylamine (101)	Good
540	Di-2-ethylhexylamine (241)	Good
148	Sec. C_{16} and C_{18} amines (tech av 255)	Ineffective
471	3, 3'-Iminobispropylamine (125)	Ineffective
492	Diethylenetriamine (103)	Ineffective
	Glyoxalidines	
269	1-Hydroxyethyl-2-heptadecenylglyoxalidine	Good
358	Glyoxalidines from wax acids	Good
	Amides and Amine Salts	
118	Amido alcohol	Good
632	Amide from 2-ethylhexoic acid and N-aminoethylmorpholine	Good
810	3, 3'-Iminobispropylamine salt of 2-ethylhexoic acid, pH 10.1	Ineffective

*52100 steel-cartridge brass at 50% RH at 77°F for 10 days.

**Numbers in parentheses are molecular weights.

Table 8

EVALUATION OF 14 COMPOUNDS BY GALVANIC TEST* IN MIL-L-6085A BASE OIL

Code No.	Compound	Result
	Sulfonates	
101	Strontium petroleum sulfonate	Good
116	Synthetic petroleum sulfonate	Good
352	Sodium dialkyl naphthalene sulfonate	Good
	Amines	
148	sec. C_{16} and C_{18} amines (tech)	Good
222	Morpholine	Good
248	t.-Octylamine	Good
492	Diethylenetriamine	Good
501	Triethylamine	Good
540	Di-2-ethylhexylamine	Good
	Amine Salts	
810	3,3'-Iminobispropylamine salt of 2-ethylhexoic acid, pH 10.1	Good
	Glyoxalidines	
269	1-Hydroxyethyl-2-heptadecenylglyoxalidine	Ineffective
358	Glyoxalidine from wax acids	Good
	Amides	
118	Amido alcohol	Good
632	Amide from N-aminoethylmorpholine and 2-ethylhexoic acid	Good

*52100 steel-cartridge brass at 50 % RH at 77°F for 10 days.

Table 9

EFFECT OF 95% RELATIVE HUMIDITY AT 100°F ON GALVANIC CORROSION INHIBITION* IN AN-O-6a BASE OIL

Code No.	Compound	Result
	Oxidized Petroleum Fractions	
139	Oxidized petroleum (MW = 405-430)	Good
140	Oxidized petroleum containing lead soaps of acids	Ineffective
141	Oxidized petroleum containing methyl esters of acids	Good
196	Oxidized petroleum fraction	Good
346	Oxidized petroleum containing lead and sodium soaps of acids	Good
	Sulfonates	
189	Triethanolamine kerylbenzenesulfonate	Good
347	Sodium dialkyl naphthalene sulfonate	Good
	Esters	
125	Pentaerythritol monooleate	Good
130	Pentaerythritol monostearate	Good
	Amine Salts	
189	Triethanolamine kerylbenzenesulfonate	Good
334	Triethanolamine salt of tall oil acids	Good
466	Morpholine salt of 2-ethylhexoic acid	Good
	Amines	
162	N-Aminoethylmorpholine	Ineffective
222	Morpholine	Good
	Miscellaneous	
118	Amido alcohol	Ineffective
190	A phosphoric acid-dodecylamine condensation product	Good
269	1-Hydroxyethyl-2-heptadecenylglyoxalidine	Good

*19 days.

Table 10

EVALUATION OF 10 COMPOUNDS IN MIL-L-6085A BASE OIL
BY GALVANIC CORROSION TEST*
(95 %, RH at 100 °F)

Code No.	Compound	Result
	Sulfonates	
101	Strontium petroleum sulfonate	Good
116	Synthetic sodium petroleum sulfonate	Ineffective
	Amines	
104	Dimethylaminomethylphenol	Good
109	2,4,6-tri-(dimethylaminomethyl)phenol	Ineffective
162	N-Aminoethylmorpholine	Good
222	Morpholine	Good
	Amine Salts	
465	N-aminoethylmorpholine salt of 2-ethylhexoic acid, pH 6.7	Good
466	Morpholine salt of 2-ethylhexoic acid	Good
	Oxidized Petroleum Fractions	
144	Oxidized petroleum fraction containing lead soaps of acids	Ineffective
	Glyoxalidine	
269	1-Hydroxyethyl-2-heptadecenylglyoxalidine	Good

*19 days.

the AN-O-6a base oil and about 40 % of those evaluated in the MIL-L-6085A base oil were effective.

3.1.2.1 Amines

As was indicated by the results of the Static Water Drop test on SAE 1020 steel, the amines were not very effective in the AN-O-6a base oil. Only two of the seven were effective, morpholine and 2.5-dimethylpiperazine. N-aminopropylmorpholine; the polyamino compounds, 3,3'-iminobispropylamine and triethylenetetramine; 2,4,6-tri- (dimethylaminomethyl)phenol; and α-methylbenzylmonoethanolamine were all ineffective.

The galvanic corrosion tests indicated that in the MIL-L-6085A base oil, the amines were slightly more effective than they were in the AN-O-6a base oil, but not to the same extent as was indicated by the Static Water Drop test in the MIL-L-6085A base oil on SAE 1020 steel. Table 11 summarizes the results of the amine evaluations. Most of the effective amines were aliphatic, even though some contained aromatic nuclei. Only one of the aromatic amines, N,N-di-(β-hydroxyethyl)-m-toluidine, was effective. The N-β-hydroxypropyl-o-toluidine, however, was ineffective. This difference could be due to a greater polarity of the first mentioned compound but other aromatic amino compounds containing other groups were not effective; for example, one aromatic amino compound containing a carboxyl group, p-aminosalicylic acid, was ineffective.

As indicated before, the aliphatic amines were the most effective. No molecular-weight range seemed particularly effective since the effective amines ranged from 8 to 18 carbon atoms. Most of the amines evaluated were secondary and tertiary. Some of the most interesting data pertain to those aliphatic amines with aromatic nuclei. α-Methylbenzylamine and α-methylbenzylmonoethanolamine were effective but the α-methylbenzyldiethanolamine was ineffective, quite probably due to the presence of the additional hydroxyl (OH) group. Tri-isopropanolamine was also ineffective but the tributylamine, like the other aliphatic amines, was effective. Octylphenoxyethoxyethyldimethylamine was effective, even though o-dimethylaminomethyl-p-butyl- and p-octylphenols were not. Apparently removing the dimethylamino group from the aromatic nucleus increases the effectiveness of this amino group. Also, the influence of the aromatic nucleus in this compound is probably quite small.

3.1.2.2 Amine Salts of Carboxylic Acids

By far the largest group of compounds evaluated in this bimetallic test were the amine salts of carboxylic acids. Most of these compounds were synthesized in ARF laboratories. The results are quite interesting. In the MIL-L-6085A base oil, the twelve salts evaluated were effective (Table 12), but in the AN-O-6a base oil most of the salts evaluated were ineffective (Table 13). Only six of the twenty-three evaluated in the AN-O-6a base oil were effective. In the MIL-L-6085A base oil certain amine salts were prepared with an excess of acid and others with an excess of amine; both types were effective. Just why these particular amine salts were effective is not known. A wide variety of amines have been reacted with 2-ethylhexoic, oleic and lauric acids and all were effective. One interpretation is that these

Table 11

EVALUATION OF 20 AMINES IN MIL-L-6085A BASE OIL
BY GALVANIC CORROSION TEST*
(95 % RH at 100 °F)

Code No.	Compound	Result
105	o-Dimethylaminomethyl-p-octylphenol	Ineffective
121	t.-Alkylamines of 12-15 carbon atoms	Good
119	N-β-Hydroxypropyl-o-toluidine	Ineffective
129	o-Dimethylaminomethyl-p-butylphenol	Ineffective
133	Octylphenoxyethoxyethyldimethylamine	Good
148	Sec. C_{16} and C_{18} amines (tech)	Good
154	Mixture C_{18} tert.-alkylamines	Good
158	o-Ethylaniline	Ineffective
160	o-Aminobiphenyl	Ineffective
166	bis(2-Aminophenyl)disulfide	Ineffective
167	p-Aminosalicylic acid	Ineffective
173	Anthranilic acid	Ineffective
193	N-Ethyl-N-β-hydroxyethylaniline	Ineffective
227	Aminohydroquinone dimethyl ether	Ineffective
228	N,N-Di-(β-hydroxyethyl)-m-toluidine	Good
229	Tri-isopropanolamine	Ineffective
230	α-Methylbenzylmonoethanolamine	Good
231	α-Methylbenzylamine	Good
233	α-Methylbenzyldiethanolamine	Ineffective
248	t.-Octylamine	Good
255	Tributylamine	Good
477	2,5-Dimethylpiperazine	Good
493	Aminoethylethanolamine	Good

* 4 weeks.

Table 12

12 AMINE SALTS THAT SHOWED EFFECTIVE INHIBITION IN MIL-L-6085A BASE OIL BY THE GALVANIC CORROSION TEST*
(95 % RH at 100 °F)

Code No.	Compound
330	Dibutylammonium oleate
465	N-Aminoethylmorpholine salt of 2-ethylhexoic acid
479	Lauric acid salt of diethylenetriamine
504	Morpholine salt of 2-ethylhexoic acid (10 % mole excess amine)
505	N-Aminoethylmorpholine salt of 2-ethylhexoic (excess acid)
506	N-Aminoethylmorpholine salt of 2-ethylhexoic acid (excess amine)
507	N-Methylmorpholine salt of 2-ethylhexoic (excess acid)
509	N-Aminopropylmorpholine salt of 2-ethylhexoic (excess acid)
510	N-Aminopropylmorpholine salt of 2-ethylhexoic acid (excess amine)
511	Hydroxyethylmorpholine salt of 2-ethylhexoic acid
542	t.-Octylamine salt of 2-ethylhexoic acid
547	α-Methylbenzyldiethanolamine salt of 2-ethylhexoic acid

*4 weeks.

Table 13

EVALUATION OF 19 AMINE SALTS IN AN-O-6a BASE OIL
BY THE GALVANIC CORROSION TEST*
(95 °/o RH at 100 °F)

Code No.	Compound**	Result
412	Dodecylnicotinium oleate	Good
413	Octadecylnicotinium oleate	Ineffective
479	Lauric acid salt of diethylenetriamine	Good
481	Oleic acid salt of diethylenetriamine	Good
503	Morpholine salt of 2-ethylhexoic acid, pH 6.0	Ineffective
504	Morpholine salt of 2-ethylhexoic acid, pH 7.7	Ineffective
505	N-Aminoethylmorpholine salt of 2-ethylhexoic acid, pH 6.5	Ineffective
506	N-Aminoethylmorpholine salt of 2-ethylhexoic acid, pH 8.1	Ineffective
507	N-Methylmorpholine salt of 2-ethylhexoic acid, pH 5.8	Good
508	N-Methylmorpholine salt of 2-ethylhexoic acid, pH 6.6	Ineffective
509	N-Aminopropylmorpholine salt of 2-ethylhexoic acid, pH 7.4	Ineffective
510	N-Aminopropylmorpholine salt of 2-ethylhexoic acid, pH 8.0	Ineffective
511	N-Hydroxyethylmorpholine salt of 2-ethylhexoic acid, pH 6.0	Ineffective
512	N-Phenylmorpholine salt of 2-ethylhexoic acid, pH 4.3	Good
542	t.-Octylamine salt of 2-ethylhexoic acid, pH 6.5	Good
543	Tributylamine salt of 2-ethylhexoic acid, pH 6.9	Ineffective
544	Benzyldimethylamine salt of 2-ethylhexoic acid, pH 6.3	Ineffective
546	α-Methylbenzylamine salt of 2-ethylhexoic acid, pH 6.6	Ineffective
547	α-Methylbenzyldiethanolamine salt of 2-ethylhexoic acid, pH 6.6	Ineffective

*4 weeks.

**Each pH value given is for a 1 °/o mixture.

salts act as detergents cleansing the surface and thereby allow better absorption of the amine, acid, or salt whichever can more effectively act as an inhibitor. In the AN-O-6a base oil this could also be true, except that a smaller number are effective in this oil. Unfortunately, these amine salts can be hydrolyzed which, along with their tendency to be oxidized, reduces their efficiency as corrosion inhibitors.

In one instance an amine found to be ineffective gave an amine salt which showed effective inhibition. This amine was α-methylbenzyldiethanolamine, and the salt was that with 2-ethylhexoic acid.

In the AN-O-6a base oil, certain trends were evident. Dodecylnicotinium oleate was effective but the higher molecular weight octadecylnicotinium oleate was not. Both the lauric and the oleic acid salt of diethylenetriamine (No. 479 and 481) were effective. The N-methylmorpholine salt of 2-ethylhexoic acid with a pH 5.8 (No. 507) was effective while the same compound at a pH of 6.6 (No. 508) was not. The N-phenylmorpholine salt and t.-octylamine salts of 2-ethylhexoic acid (No. 512 and 542) were effective. The amines from which these three effective salts were prepared were not as basic as some of the other amines mentioned in Table 13. Of the three, the t.-octylamine should be the most basic. Most of the other monoamines and the morpholine derivatives should be more basic and the salts of these were ineffective. These results seem to indicate that salts of weak monoamines and low basicity morpholines are the most effective inhibitors against this bimetallic system in AN-O-6a base oil under the conditions mentioned.

3.1.2.3 Oxidized Petroleum Fractions

Ten oxidized petroleum fractions and derivatives were evaluated in MIL-L-6085A base oil; the results are given in Table 14. Six of these were found to be effective and four were not. Low molecular-weight products and those containing lead soaps of acids were ineffective. While only meager data is available concerning the exact nature of the products tested, it can be at least tentatively concluded that if the molecular weights are high enough, the products as obtained and those having methyl esters of the acids present are effective inhibitors. Other characteristics of this type of material, such as compatibility and low-temperature properties, however, make them less interesting for corrosion-inhibiting formulations.

Only two compounds were evaluated in the AN-O-6a base oil; No. 196 was effective while No. 137 (MW = 450 - 490) was ineffective.

3.1.2.4 Esters

Of the eleven esters evaluated in the MIL-L-6085A base oil only two were effective, namely, petaerythritol monooleate and pentaerythritol monostearate. These results seem to indicate that additional hydroxyl groups are important or that hydroxymethyl groups contribute to the effectiveness. Another polyhydroxy compound, polyoxyethylene sorbitan monostearate, was ineffective but it does not contain the hydroxymethyl groups. Other esters of polyglycols were also ineffective. The two phosphite esters evaluated were also ineffective. The results are shown in Table 15.

Table 14

EVALUATION OF CERTAIN OXIDIZED PETROLEUM FRACTIONS AND DERIVATIVES IN MIL-L-6085A BASE OIL BY GALVANIC CORROSION TEST*
(95 % RH at 100 °F)

Code No.	Compound	Result
136	An oxidized petroleum fraction containing methyl esters of acids	Good
137**	Oxidized petroleum fraction (MW = 405 - 490)	Good
138	Oxidized petroleum fraction	Good
139	Oxidized petroleum fraction	Ineffective
141	Oxidized petroleum containing methyl esters of acids	Good
142	Organic acids from oxidized petroleum fraction (MW = 450 - 490)	Good
143	Derivatives of oxidized petroleum fractions	Good
144	Oxidized petroleum fraction containing lead soaps of acids	Ineffective
196***	Hydrocarbon oil oxidation products	Ineffective
346	Oxidized petroleum fraction containing lead and sodium derivatives of acids present	Ineffective

*4 weeks.

**Ineffective in AN-O-6a base oil.

***Also effective in AN-O-6a base oil.

Table 15

EVALUATION OF CERTAIN ESTERS IN MIL-L-6085A BASE OIL
BY GALVANIC CORROSION TEST*
(95 °/o RH at 100 °F)

Code No.	Compound	Result
112	Diethyleneglycol oleate	Ineffective
113	Triethyleneglycol di-2-ethylbutyrate	Ineffective
115	Triethyleneglycol di-2-ethylhexoate	Ineffective
117	Polyoxyethylene ester of tall oil acids	Ineffective
122	Triphenyl phosphite	Ineffective
124	Butyl p-hydroxybenzoate	Ineffective
125	Pentaerythritol monooleate	Good
130	Pentaerythritol monostearate	Good
151	Phosphite ester of an alkylated phenol	Ineffective
153	Polyoxyethylene sorbitan monostearate	Ineffective
172	Propyl gallate	Ineffective

*4 weeks.

Only three esters were evaluated in the AN-O-6a base oil, and the two containing the pelargono radical were effective. These were allyl α-pelargono propionate and octyl polylactate pelargonate; 2-ethylhexylpolylactate-2-ethylhexanoate was ineffective.

3.1.2.5 Sulfonates

Ten sulfonates were evaluated in the AN-O-6a base oil and nine, in the MIL-L-6085A base oil. In most instances complete commercial information concerning the sulfonates was not available so that final conclusions can not be drawn.

The products evaluated in AN-O-6a base oil are summarized in Table 16. The products of higher molecular weight appeared to be less effective in this test than those of lower molecular weight. Compound 185, the waxyl benzenesulfonate, for example, was ineffective. Compound 239 is a high-molecular-weight sulfonate which has found considerable use in corrosion-inhibiting formulations, but it was ineffective. On the other hand, the keryl-benzenesulfonate, where keryl is approximately 12 carbon atoms was effective as the triethanolamine salt. It is also of interest that the sodium salt of di-alkyl (probably dinonyl) naphthalene sulfonate (No. 347) was effective while the barium salt (No. 351) was not. The other sulfonates were restricted compositions hence no information is available to suggest which of their characteristics might be the effective ones.

In the MIL-L-6085A base oil five of the nine sulfonates evaluated were effective; the results are summarized in the Table 17. Very little can be

Table 16

EVALUATION OF 10 SULFONATES IN AN-O-6a BASE OIL BY GALVANIC CORROSION TEST*
(95 % RH at 100 °F)

Code No.	Compound	Result
116	Synthetic sodium petroleum sulfonate	Good
185	Sodium waxylbenzene sulfonate	Ineffective
189	Triethanolamine kerylbenzene sulfonate	Good
239	Sulfonated high-molecular-weight organic base	Ineffective
322	Sulfonated petroleum product, butyl alcohol, and selected mineral oils	Good
347	Synthetic sodium alkyl aryl sulfonate	Good
348	Synthetic petroleum sulfonate	Ineffective
349	High-molecular-weight sodium petroleum sulfonate	Ineffective
351	Barium dialkyl naphthalene sulfonate	Ineffective
352	Sodium dialkyl naphthalene sulfonate	Good

*4 weeks.

Table 17

EVALUATION OF NINE SULFONATES IN MIL-L-6085A BASE OIL BY GALVANIC CORROSION TEST*
(95 % RH at 100 °F)

Code No.	Compound	Result
116	Synthetic sodium petroleum sulfonate	Good
131	Petroleum sodium sulfonate	Ineffective
135	Synthetic petroleum sulfonate	Ineffective
185	Mixed molecular weight sodium petroleum sulfonate	Ineffective
189	Triethanolamine salt of kerylbenzene sulfonate	Ineffective
347	Sodium dialkyl naphthalene sulfonate	Good
348	Synthetic sodium petroleum sulfonate	Good
351	Barium dialkyl naphthalene sulfonate	Good
523	Petroleum sodium sulfonate	Good

*4 weeks.

deduced from the MIL-L-6085A base oil results because of incomplete data on the products tested. The barium dialkyl naphthalene sulfonate which was found to be ineffective in AN-O-6a base oil was effective in MIL-L-6085A base oil.

3.1.2.6 Glyoxalidines

Some data are available on certain glyoxalidines in AN-O-6a base oil. 1-Hydroxyethyl-2-heptadecenylglyoxalidine (No. 269) was effective in the AN-O-6a base oil, as was the 1-aminoethyl derivative (No. 481). Similarly, 1-aminoethyl-2-undecylglyoxalidine (No. 479) was also effective. The high-molecular-weight glyoxalidine (No. 358), believed to be a 1-amino substituted product, was ineffective.

The 1-hydroxyethyl-2-heptadecenylglyoxalidine (No. 269) and the 1-aminoethyl-2-undecylglyoxalidine were also effective in MIL-L-6085A base oil. The 1-hydroxyethyl-2-undecyl and the 1-aminoethyl-2-(-8-heptadecenyl) were ineffective; these failures indicate an effect of the amino and hydroxy groups when coupled with high molecular weight.

Alcohols, phenols, amides, sulfides, sulfates, nitriles and acids evaluated in the MIL-L-6085A base oil were all ineffective. Because t.-hexadecyl mercaptan was effective but the t.-dodecyl mercaptan was not, a molecular weight effect seems to be indicated. The amides evaluated were also found to be ineffective in the AN-O-6a base oil.

3.1.3 95 % Relative Humidity at 160 °F for 3 weeks

In the AN-O-6a base oil 90 compounds were evaluated under these conditions and in MIL-L-6085A base oil over 160. These conditions are more severe than those previously described, and it was thought that a better selection of effective products would result. Only two compounds were found to be effective in the AN-O-6a base oil, namely, No. 141, an oxidized petroleum fraction containing the methyl esters of the acids, and No. 466, the morpholine salt of 2-ethylhexoic acid with a pH of 6.7. Compound 141 was not evaluated at 95 % RH at 100 °F for 4 weeks. Other oxidized petroleum fractions and derivatives were evaluated at 95 % RH at 160 °F and all were ineffective. The same is true for many carboxylic acid amine salts.

Seven compounds were found to be effective under these conditions in the MIL-L-6085A base oil and four of the seven were sulfonates. The compounds which were effective are summarized in Table 18.

With all these compounds with the exception of No. 348, a slight discoloration of the brass clip occurred during the test period. The strontium sulfonate (101) was effective at 50 % RH at 77 °F. Compound 322 had not been previously tested in MIL-L-6085A base oil. Compound 348 was satisfactory at 95 % RH at 100 °F for 4 weeks, and No. 352 was effective at 50 % RH at 77 °F. In this test the strontium salt of the particular sulfonic acid used was effective although the sodium salt of the same sulfonic acid (No. 185) was ineffective.

To summarize the results of all the galvanic system tests, it can be stated that the sulfonates, the aliphatic amines, certain amine salts of car-

Table 18

SEVEN EFFECTIVE COMPOUNDS IN MIL-L-6085A BASE OIL BY GALVANIC CORROSION TEST*
(95 % RH at 160 °F)

Code No.	Compound
	Sulfonates
101	Strontium sulfonate
322	Sulfonated petroleum products, butyl alcohol and selected mineral oils
348	Synthetic sodium petroleum sulfonate
352	Sodium dialkyl naphthalene sulfonate
	Amines
471	3, 3'-Iminobispropylamine
	Acid
232	2-Ethylhexoic acid
	Mercaptan
114	t.-Hexadecyl mercaptan

*3 weeks.

boxylic acids, the high-molecular-weight oxidized petroleum fractions, the petroleum fractions containing the methyl esters of the acids, and the esters of fatty acids with pentaerythritol were effective inhibitors in the MIL-L-6085A base oil. Alcohols, acids, amides, sulfates, phenols, sulfides and nitriles tested were ineffective.

With the AN-O-6a base oil, the effective compounds were the sulfonates of lower molecular-weight, the aliphatic amines, the carboxylic acid salts of amines, polyamines, and certain N-substituted alkyl or aryl morpholines; the glyoxalidines; and a few esters containing a pelargonic group.

3.2 Effects of Amine-Sulfonate Combinations on SAE 1020 Steel

For convenience of discussion the amine salts, the compounds other than the sulfonates, listed in Table 2 may be divided into two groups. A compound that was a good inhibitor in a 0.05 % solution (see the third column of the table) is in Class A while a compound that was a fair or ineffective inhibitor in a 0.05 % solution is in Class B. From the data in Table 2, No. 255, tributylamine cannot be classified as either A or B.

Although a 0.1 % solution of barium dialkyl naphthalene sulfonate (No. 351) is only a fair inhibitor, 0.05 % of No. 351 with 0.05 % concentration of any compound in Class B except No. 776, sorbitan mono-oleate) improves the inhibiting qualities to good. Without No. 351 a Class B compound (with the exception of 776) was a good inhibitor in a 0.2 % solution. Thus, by reducing the concentration of the Class B inhibitor to 0.05 % and supplementing with 0.05 % of No. 351, it was possible to obtain an effectiveness equal to a 0.2 % Class B concentration. However, a 0.2 % solution of No. 351 alone was still only a fair inhibitor.

Any combination of No. 351 with a Class A compound was still a good inhibitor.

At this time it is not possible to interpret the corrosion mechanism of the effective amines and amine salts from the limited data available. It is possible that Class A compounds would be effective in more dilute solutions. Also, in a combination solution a lower concentration of No. 351 and/or the Class B compound might still be effective.

Although the synthetic petroleum sulfonate, No. 116, alone was a better inhibitor than No. 351 alone, in combinations it was not as effective.

3.3 JAN-H-792 Cabinet Tests

The results of the 100-hr JAN-H-792 cabinet tests indicate that two general types of compounds were effective in MIL-L-6085A base oil. These were sulfonates and amine salts of oleic and linoleic acid.

Only 12 of the 235 compounds tested with the cabinet were effective; these are all listed in Table 19. Certain salts of pelargonic acid almost passed the test.

Only the three sulfonates listed in Table 19 were effective. However, several were effective to 90 hr. The inhibiting effect of strontium petroleum

Table 19

COMPOUNDS EFFECTIVE AFTER 100 HOURS IN JAN-H-792 CABINET TEST (5.0 % concentration)

Code No.	Compound
101*	Strontium petroleum sulfonate
322	Sulfonated petroleum products, butyl alcohol, and selected mineral oils
351	Barium dialkyl naphthalene sulfonate
585*	N-Aminoethylmorpholine salt of oleic acid, pH 8.8
595*	Tetraethylenepentamine salt of oleic acid, pH 10.3
596	α-Methylbenzylamine salt of oleic acid, pH 8.6
600	α-Methylbenzylmonoethanolamine salt of oleic acid, pH 8.4
606	N-Aminopropylmorpholine salt of oleic acid**
611	t.-Octylamine salt of oleic acid**
614*	Tetraethylenepentamine salt of oleic acid**
649*	3,3'-Iminobispropylamine salt of linoleic acid**
651	N-Aminopropylmorpholine salt of linoleic acid**

*5 % equivalent not completely soluble.

**Heated at 150°C for 3 hr.

sulfonate must be associated with the strontium because the sodium salt of the same sulfonic acid had failed in 24 hr.

Apparently oleic or linoleic acid was a necessary part of those amine salts which were effective. Many other monocarboxylic and dicarboxylic acid salts of amines failed the test. Nine amine salts of oleic or linoleic acid were effective (Table 19) and most of the compounds that did not quite pass the tests were amine salts of linoleic or oleic acid.

Nine formulations of amine salts (Table 20) in combination with a sulfonate and an oxidation inhibitor were tested in MIL-L-6085A base oil. All nine formulations contained 5.0 °/₀ barium dialkyl naphthalene sulfonate and 0.2 °/₀ phenothiazine. A third, and differing, constituent was 2.0 °/₀ of an amine salt (2.4 °/₀ in No. 906); all were amine salts of a carboxylic acid, except the 3,3'-iminobispropylamine salt of a mahogany sulfonic acid in No. 905.

The six amine salts of oleic or linoleic acid passed the tests, and the other three almost passed. However, it must be recognized that the amine salts are likely to be less stable to hydrolysis and it is quite possible that a 0.2 °/₀ concentration of phenothiazine would not prevent oxidation to the extent necessary.

Furthermore, phenothiazine does have a negative corrosion inhibiting effect. A 5 °/₀ solution of barium dialkyl naphthalene sulfonate (No. 351 in Table 19) was an effective inhibitor but panels tested with No. 902, which contained 0.2 °/₀ phenothiazine and 5 °/₀ barium dialkyl naphthalene sulfonate, corroded in 72 hr.

SECTION IV

SUMMARY AND CONCLUSIONS

An extensive survey was made with the JAN-H-792 humidity cabinet test to find polar organic compounds that would pass the 100-hr requirements of this test. Before evaluating these materials it was necessary to correlate the ARF cabinet with the cabinets at Wright Field. The investigation indicated that differences were primarily due to differences in air flow rates and air volumes because the porous dispersers have different porosities in the various cabinets. By using a wet test meter to adjust air flows to those of the test data obtained at Wright Field, correlation was achieved. It was also shown that differences in specimen preparation techniques did not affect the results.

Over two hundred compounds and several formulations were evaluated by this method. Of the different types of compounds evaluated only two, sulfonates and amine salts of oleic and linoleic acid were effective, and not all compounds of these types were effective. Three sulfonates (strontium petroleum sulfonate, sulfonated petroleum products in a butyl alcohol mineral oil solution, and barium dialkyl naphthalene sulfonate) were effective. The oleic acid salts of N-aminoethylmorpholine, tetraethylenepentamine, α-methylbenzylamine and α-methylbenzylmonoethanolamine were effective. After heating at 150 °C for 3 hr, the aminopropylmorpholine, the t.-octylamine, and the tetraethylenepentamine salts of oleic acid were effective. Similarly, the

Table 20

FORMULATIONS OF NINE AMINE SOLUTIONS
TESTED IN JAN-H-792 CABINET

All solutions contained	%
Barium dialkyl naphthalene sulfonate	5.0
Phenothiazine*	0.2

Formulation	2% of Third Constituent — Compound	Code
Passed Test		
907	N-Aminopropylmorpholine salt of linoleic acid**	651
908	N-Aminoethylmorpholine salt of oleic acid	585
909	α-Methylbenzylamine salt of oleic acid	596
910	α-Methylbenzylmonoethanolamine salt of oleic acid	600
912	Tetraethylenepentamine salt of linoleic acid	645
914	3,3'-Iminobispropylamine salt of linoleic acid**	649
Failed Test		
905	3,3'-Iminobispropylamine salt of a mahogany sulfonic acid	818
906	m-Aminobenzotrifluoride salt of pelargonic acid**	694***
911	Morpholine salt of oleic acid	583

*Crude material purchased from Eastman Kodak and recrystallized.

**Heated at 150°C for 3 hr.

***2.4% conc.

N-aminopropylmorpholine and the 3,3'-iminobispropylamine salts of linoleic acid were effective after heating for 3 hr at 150 °C.

Six amine salt formulations were also effective. Each of the successful formulations contained 5 % barium naphthalene sulfonate and 0.2 % phenothiazine as oxidation inhibitor. Each formula in addition contained one of the following amine salts.

Code	
907	N-Aminopropylmorpholine salt of linoleic acid*
908	N-Aminoethylmorpholine salt of oleic acid
909	α-Methylbenzylamine salt of oleic acid
910	α-Methylbenzylmonoethanolamine salt of oleic acid
912	Tetraethylenepentamine salt of linoleic acid
914	3,3'-Iminobispropylamine salt of linoleic acid*

These results indicate that the presence of an unsaturated linkage in the carboxylic acid is necessary for inhibition by amine salts in the JAN-H-792 cabinet and, also, that certain sulfonates are quite effective. The barium dialkyl naphthalene sulfonate plus phenothiazine in the diester oil was ineffective at 72-hr test duration. Each of these amine salts was effective at 5 % concentration but only 2 % was needed in the formulation to achieve the same inhibiting results. Adding phenothiazine to a 5 % solution of barium dialkyl naphthalene sulfonate had an adverse effect since this composition (No. 902) failed, whereas the composition without the phenothiazine (No. 351) passed.

A series of combinations of polar organic compounds were evaluated by the Static Water Drop test. The common ingredient in each of three series of tests were sodium dialkyl naphthalene sulfonate, barium dialkyl naphthalene sulfonate, and a synthetic petroleum sulfonate. These were only fair inhibitors at 0.1 % concentration. In combination with various amines and amine salts of 0.5 % concentration each, increased effectiveness was achieved since most of the amines and amine salts were ineffective or only fair at 0.05 % concentrations.

The mechanism of corrosion inhibition as related to structural and functional group effects was also studied on a bimetallic system at different conditions of relative humidity and temperature. The largest study was made on nearly 200 compounds at 95 % RH at 100 °F in the American Instrument Company cabinet. In the MIL-L-6085A base oil, aliphatic polar organic compounds were effective whereas materials containing aromatic nuclei were generally ineffective. Only a few aromatic-containing compounds were effective. Certain heterocyclic amino compounds and derivatives were also effective. Specifically the alkyl aryl sulfonates, aliphatic amines, certain amine salts of carboxylic acids, high molecular weight oxidized petroleum fractions and derivatives, especially methyl esters, and certain fatty acid esters of polyhydroxymethyl compounds were effective. Alcohols, acids, amides, sulfates, phenols, sulfides and nitriles were ineffective.

These results indicate the sulfonic acid groups are effective if attached to the right alkyl aryl hydrocarbon radical. One such radical specifically

---

*Heated at 150 °C for 3 hr.

known to be effective is dinonyl naphthalene. A few other sulfonates are effective but their composition is unknown.

Amine effectiveness is not a function of molecular weight since the amines which proved to be effective range between 8 and 18 carbon atoms. Most of those evaluated were those with an amino group on a secondary or tertiary carbon atom and these proved to be effective. Certain aliphatic amines containing an aromatic nucleus were effective; in most instances these were primary amines. In one effective compound one of the amino hydrogens was substituted by a hydroxyethyl group. The presence of two hydroxyethyl groups on the same nitrogen gives an ineffective compound. If a dimethylaminomethyl group is attached to an aromatic ring, the compound is ineffective. A dimethylaminomethyl group at the end of an aliphatic chain attached to an aromatic nucleus results in an effective inhibitor.

Amine salts of 2-ethylhexoic, lauric and oleic acids were effective. Most of the effective salts were from polyamino compounds including N-substituted aminoalkyl morpholines.

In the AN-O-6a base oil salts of lower basicity amines such as N-methyl and N-phenyl morpholine were effective.

The high-molecular-weight oxidized petroleum fractions were effective by themselves and in combination with methyl esters of the acids. Lead soaps were ineffective.

In general, the esters were not effective; only two showed good inhibition and these were two fatty acid esters of pentaerythritol.

1-Hydroxyethyl-2-heptadecenylglyoxalidine was effective in both oils. Substituting an aminoethyl in the 1-position of this compound reduced effectiveness.

At a relative humidity of 95% at 160°C for a 3-week period, the most effective inhibitors in MIL-L-6085A base oil were the alkyl aromatic sulfonic acid derivatives.

APPENDIX

LABORATORY SYNTHETIC PROCEDURES

5.1 Condensation Reactions

The yields of these condensation reactions are not listed, since they were sacrificed in the interests of purity.

5.1.1 4,5-Dimorpholinocatechol*

A solution of 44 g (0.4 M) catechol and 70 g (0.8 M) morpholine in 500 ml EtOH was refluxed for 11-1/2 hr during which time a slow stream of air was bubbled into the solution. The alcohol was evaporated from the reddish-brown solution by means of a water bath, until the total volume was concentrated to 250-300 ml. The concentrated solution was cooled in an ice-bath and filtered. The yield was a dark product. After two recrystallizations from EtOH, the resulting very light tan solid melted at 218-220 °C (uc) with decomposition (Fisher-Johns apparatus); literature mp = 222°. The solid turns a reddish-brown on exposure to air.

5.1.2 Attempted Condensations Between Aminoethylmorpholine and Aminoethyl Bromide Hydrobromide

In a round-bottomed flask fitted with a reflux condenser were placed 13.0 g (0.1 M) N-aminoethylmorpholine, 22.5 g (0.11 M) aminoethyl bromide hydrobromide, 25.2 g (0.3 M) $NaHCO_3$, and sufficient ethanol to dissolve the organic components. The mixture was refluxed for about 10 hr, after which time no apparent change had taken place.

The alcohol was evaporated from the mixture, 1 g copper-bronze powder was added and the mixture was refluxed 8 hr, by which time the liquid portion had darkened. The reaction mixture was cooled, treated with cold, concentrated sodium hydroxide solution, and extracted with ether. The ethereal solution was dried over Drierite. The solution was filtered, the ether was evaporated, and the residue was distilled under reduced pressure through an open, vacuum-jacketed, still head. A pale yellow oil (6.4 g) was collected gradually over the range 94-124°/17-18 mm leaving a small amount of dark residue. Apparently no significant amount of condensation occurred between the reactants under these conditions.

5.2 Mannich Reactions

5.2.1 2,4,6-Tri(morpholinomethyl)phenol**

In a three-necked flask fitted with stirrer, thermometer, and adding funnel--were placed 47 g (0.5 M) phenol and 153 g (1.75 M) morpholine. To the solution, with stirring and cooling to 20-25°, was added 139 g (1.65 M) of 36 % formalin solution. The turbid solution obtained was stirred for 2 hr at

*Henry and Dehn, J. Am. Chem. Soc. 74, 278, 1952.

**Bruson and MacMullen. J. Am. Chem. Soc. 63, 270, 1941.

20-25° and then heated for 1 hr at 85-90° (heating mantle), with the adding funnel replaced by an air condenser. The resulting clear solution was concentrated on the aspirator until all the water was removed. The viscous residue, which slowly crystallized on standing several days, was crystallized from ethyl acetate and recrystallized from an excess of ether. The white solid melted at 105-106.5° (uc)(Fisher-Johns apparatus); literature mp = 106-107°.

5.2.2 Reaction Product of Hydroquinone and Morpholine*

In a three-necked flask fitted with stirrer, thermometer, and adding funnel--were placed 55.0 g (0.5 M) hydroquinone, 130.8 g (1.5 M) morpholine and 130 ml water. The mixture was immediately cooled and maintained at 20-25°C with stirring, while 126.5 g (1.5 M) of 36 % formalin solution was added, dropwise. The resulting red solution was stirred 1 hr at 20-25° and then slowly heated (heating mantle) to 80° over a period of 2 hr, during which time a fine tan precipitate slowly appeared. After standing overnight at room temperature, the mixture was filter, washed with water and warm ethanol by decantation, and air-dried. The tan-solid was dissolved in the minimum amount of hot benzene, cooled somewhat and precipitated with an excess of benzene. The resulting white, micaceous flakes were filtered, air-dried, and recrystallized from a saturated solution in benzene. A portion of the air-dried white micaceous flakes were transformed to a dull white solid on heating to 80°; the bulk of the solid was then heated in an oven at 95-100° for 1 hr. The white solid obtained, melted at 204.5-206.5 (uc) with some decomposition (Fisher-Johns apparatus).

*Caldwell and Thompson. ibid, 61, 765, 1939.

Table 21

CODE FOR CHEMICALS TESTED

Code	Manufacturer's Designation	Manufacturer
101	Strontium Petronate	Sonneborn
104	DMP-10	Rohm and Haas
105	DMP-18	Rohm and Haas
109	DMP-30	Rohm and Haas
112	Diglycol Oleate S	Glyco Products
113	Flexol Plasticizer 3GH	Carbide and Carbon
114	tert.-Dodecyl (3B) Mercaptan	Sharples
115	Flexol Plasticizer 3GO	Carbide and Carbon
116	Synfonate 26	U. B. Bray
117	Renex 48	Atlas Powder Company
118	Ultrapone DL	Ultra Chemical Company
119	N-β-Hydroxypropyl-o-toluidine	Tennessee Eastman
121	Alkylamine 81	Rohm and Haas
122	Triphenyl Phosphite	Monsanto
124	Butyl Parasept	Heyden
125	Pentamull 126	Heyden
129	DMP-14	Rohm and Haas
130	Pentawax 145	Heyden
131	Carchex 200	Carlisle
133	Octylphenoxyethoxyethyldimethylamine	Rohm and Haas
135	Synfonate 9P	U. B. Bray
136	Alox 152	Alox Corporation
137	Alox 125	Alox Corporation
138	Alox 102	Alox Corporation
139	Alox 100	Alox Corporation
140	Alox 707	Alox Corporation
141	Alox 350	Alox Corporation
142	Alox 325	Alox Corporation
143	Alox 311	Alox Corporation
144	Alox 701	Alox Corporation
148	Armeen 2HT	Armour and Company
151	Santolube 31	Monsanto

Table 21 (cont)

Code	Manufacturer's Designation	Manufacturer
153	Tween 61	Atlas Powder Company
154	Alkylamine JM-R	Rohm and Haas
158	o-Ethylaniline	Monsanto
160	o-Aminobiphenyl	Monsanto
162	N-Aminoethylmorpholine	Carbide and Carbon
166	bis(2-Aminophenyl) Disulfide	American Cyanamid
167	p-Aminosalicylic Acid	Monsanto
172	Propyl Gallate	Heyden
173	Anthranilic Acid	Dow
174	1,3-Diethylthiourea	Sharples
175	1,3-Diisopropylthiourea	Sharples
176	Phenylbiguanide	American Cyanamid
178	Tergitol Penetrant 08	Carbide and Carbon
185	Petronate	Sonneborn
189	Triethanolamine Kerylbenzenesulfonate	Wyandotte
190	Victamine C	Victor Chemical Company
193	N-Ethyl-N, β-hydroxyethylaniline	Tennessee Eastman
194	Tetraethylenepentamine	Carbide and Carbon
196	Alox 600	Alox Corp.
200	Pluracol 0-4018	Wyandotte
201	Pluracol 0-4029	Wyandotte
205	Pluracol 0-3886	Wyandotte
206	Pluracol 0-3993	Wyandotte
209	Pluracol 0-3193	Wyandotte
214	Pluracol 0-3803	Wyandotte
216	Pluracol 0-3917	Wyandotte
222	Morpholine	Carbide and Carbon
226	Neo-Fat D-142	Armour and Company
227	Aminohydroquinone Diethyl Ether	Tennessee Eastman
228	N,N-Di(β-hydroxyethyl)-m-toluidine	Tennessee Eastman
229	Tri-isopropanolamine	Carbide and Carbon
230	α-Methylbenzylmonoethanolamine	Carbide and Carbon
231	α-Methylbenzylamine	Carbide and Carbon

Table 21 (cont)

Code	Manufacturer's Designation	Manufacturer
232	2-Ethylhexoic Acid	Carbide and Carbon
233	α-Methylbenzyldiethanolamine	Carbide and Carbon
237	CP-617	Monsanto
239	Hiboss	Monsanto
246	Dinonylamine	Rohm and Haas
247	Benzyldimethylamine	Rohm and Haas
248	t.-Octylamine	Rohm and Haas
255	Tributylamine	Sharples
269	Amine 220	Carbide and Carbon
293	DPG	duPont
308	Trimene Base	Naugatuck
317	VGB	Naugatuck
322	Bondogen	Vanderbilt
330	Barak	duPont
334	Ridacto	Spencer
346	Alox L-1673	Alox Corporation
347	Nasul	Vanderbilt
348	Synfonate 36W	U. B. Bray
349	Pentronate-H	Sonneborn
351	Barium Alkyl Aryl Sulfonate	King Organic Chemicals
352	Sodium Alkyl Aryl Sulfonate	King Organic Chemicals
357	Amine C	Alrose Chemical Company
358	Amine O	Alrose Chemical Company
412	Dodecylnicotinium Oleate	ERRL - USDA
413	Octadecylnicotinium Oleate	ERRL - USDA
465	3602C1644	ARF
466	3601C1644	ARF
471	3,3'-Iminobispropylamine	Carbide and Carbon
473	N-Aminopropylmorpholine	Carbide and Carbon
475	N-Hydroxyethylmorpholine	Carbide and Carbon
477	2,5-Dimethylpiperazine	Carbide and Carbon
478	Petrobase 210	Penn. Refining Company
479	0301C1840	ARF

Table 21 (cont)

Code	Manufacturer's Designation	Manufacturer
481	4701C1644	ARF
482	0901C1840	ARF
490	Hydrofuramide	Quaker Oats Co.
491	Triethylenetetramine	Carbide and Carbon
492	Diethylenetriamine	Carbide and Carbon
493	Aminoethylethanolamine	Carbide and Carbon
497	32C1644	ARF
499	3204C1644	ARF
501	Triethylamine	Sharples
503	2703C1840	ARF
504	2704C1840	ARF
505	2705C1840	ARF
506	2706C1840	ARF
507	2602C1840	ARF
508	2603C1840	ARF
509	2702C1840	ARF
510	2701C1840	ARF
511	2604C1840	ARF
512	2601C1840	ARF
523	Petroleum Sulfonate	Shell Chemical Company
540	Di-2-ethylhexylamine	Carbide and Carbon
542	4602C1840	ARF
543	4603C1840	ARF
544	4604C1840	ARF
546	4606C1840	ARF
547	4607C1840	ARF
563	0101C2102	ARF
564	0102C2102	ARF
565	0201C2102	ARF
566	0202C2102	ARF
567	0203C2102	ARF
569	0205C2102	ARF
570	0412C2102	ARF
571	0607C2102	ARF

Table 21 (cont)

Code	Manufacturer's Designation	Manufacturer
572	0608C2102	ARF
573	0609C2102	ARF
574	0610C2102	ARF
576	0612C2102	ARF
577	0613C2102	ARF
582	0312C2102	ARF
583	0206C2102	ARF
584	0301C2102	ARF
585	0207C2102	ARF
587	0209C2102	ARF
588	0210C2102	ARF
592	0302C2102	ARF
594	0304C2102	ARF
595	0305C2102	ARF
596	0306C2102	ARF
600	0310C2102	ARF
601	0311C2102	ARF
602	0214C2102	ARF
603	0215C2102	ARF
604	0216C2102	ARF
605	0217C2102	ARF
606	0218C2102	ARF
610	0401C2102	ARF
611	0402C2102	ARF
613	0404C2102	ARF
614	0405C2102	ARF
615	0406C2102	ARF
619	0410C2102	ARF
620	0411C2102	ARF
626	β-Phenylethylamine	Monsanto
632	β-(2-Ethylhexamido)-N-ethylmorpholine	ARF
633	0501C2102	ARF
635	0503C2102	ARF

Table 21 (cont)

Code	Manufacturer's Designation	Manufacturer
636	0504C2102	ARF
642	0510C2102	ARF
643	0511C2102	ARF
644	0512C2102	ARF
645	0513C2102	ARF
646	0514C2102	ARF
649	0603C2102	ARF
650	0605C2102	ARF
651	0605C2102	ARF
652	0606C2102	ARF
656	0704C2102	ARF
657	0705C2102	ARF
658	0706C2102	ARF
659	0707C2102	ARF
660	0708C2102	ARF
662	0710C2102	ARF
663	0711C2102	ARF
664	0712C2102	ARF
665	0713C2102	ARF
666	0714C2102	ARF
669	0717C2102	ARF
671	0719C2102	ARF
672	0720C2102	ARF
673	0721C2102	ARF
674	0722C2102	ARF
677	0803C2102	ARF
678	0804C2102	ARF
679	0805C2102	ARF
680	0806C2102	ARF
681	0807C2102	ARF
682	0808C2102	ARF
683	0809C2102	ARF
684	0810C2102	ARF
685	0811C2102	ARF

Table 21 (cont)

Code	Manufacturer's Designation	Manufacturer
689	0815C2102	ARF
690	0816C2102	ARF
691	0817C2102	ARF
692	0818C2102	ARF
693	0819C2102	ARF
694	0820C2102	ARF
695	0821C2102	ARF
696	0822C2102	ARF
697	0823C2102	ARF
698	0824C2102	ARF
701	0903C2102	ARF
711	0913C2102	ARF
720	1003C2102	ARF
730	1013C2102	ARF
738	Ethylenediamine	Carbide and Carbon
739	Propylenediamine	Carbide and Carbon
742	Emulsifier STH	General Aniline and Film Corporation
744	Cyclohexylamine salt of Emulsifier STH	General Aniline and Film Corporation
747	0517C2102	ARF
748	0518C2102	ARF
749	0519C2102	ARF
753	2902C2102	ARF
757	2906C2102	ARF
764	2806C2102	ARF
765	2807C2102	ARF
773	2914C2102	ARF
774	2915C2102	ARF
776	Span 80	Atlas Powder Company
783	Barium Sulfonate	Bryton, Incorporated
784	Calcium Sulfonate	Bryton, Incorporated
785	Strontium Sulfonate	Bryton, Incorporated
790	2002C2298	ARF

Table 21 (cont)

Code	Manufacturer's Designation	Manufacturer
791	2003C2298	ARF
792	2004C2298	ARF
793	2005C2298	ARF
794	2006C2298	ARF
796	2008C2298	ARF
797	2009C2298	ARF
798	2010C2298	ARF
799	2011C2298	ARF
800	2012C2298	ARF
801	2013C2298	ARF
803	2101C2298	ARF
805	2103C2298	ARF
806	2104C2298	ARF
807	2105C2298	ARF
810	2201C2298	ARF
812	n-Butylarsonic Acid	Eastman Kodak
818	4603C2298	ARF
819	4604C2298	ARF
820	4901C2298	ARF
823	4904C2298	ARF
826	4201C2037	ARF
827	4101C2037	ARF
843	DAK	Mathieson Company, Inc.
845	Duomeen S	Armour and Company
846	Duomeen T	Armour and Company
847	Duomeen 12	Armour and Company
848	Duomeen C	Armour and Company
849	2-Amino-n-octane	Eastman Kodak
851	0802C2580	ARF
852	Monoethylamine	Sharples
853	Diethylamine	Sharples
854	Dibutylamine	Sharples
856	Di-iso-propylamine	Sharples
857	n-Heptylamine	Mathieson Company, Inc.

Table 21 (cont)

Code	Manufacturer's Designation	Manufacturer
858	n-Octylamine	Mathieson Company, Inc.
860	n-Decylamine	Mathieson Company, Inc.
862	Stearic Acid	Hormel Foundation
863	Linoleic Acid	Hormel Foundation
864	Monopropylamine	Sharples
865	Dipropylamine	Sharples
866	Tripropylamine	Sharples
867	sec.-Butylamine	Sharples
868	iso-Butylamine	Sharples
869	Di-iso-butylamine	Sharples
870	sec.-Amylamine	Sharples
875	4201C2496	ARF
876	4301C2496	ARF
877	Elaidic Acid	ERRL-USDA
878	Winsor Lube L-245X	F.E. Anderson Oil Co.
879	1701C2580	ARF
880	4701C2496	ARF
881	4501C2496	ARF
882	4301C2496	ARF
883	2001C2580	ARF
884	PO 10K-2	Eclipse-Pioneer, Div. of Bendix Aviation
885	Cosmolubric No. 270	E.F. Houghton Company
886	L-503 (Base Stock)	Lehigh
887	Aerolube 51	American Cyanamid
888	2501C2580	ARF
889	3301C2580	ARF
892	Univis P-38	Esso
894	PO 10K-3	Eclipse-Pioneer Div. of Bendix Aviation
895	β-(2-Pentadecyl-2-imidazolinyl-1)-ethanol	U. of New Mexico
896	β-(2-Hendecyl-2-imidazolinyl-1) ethanol	U. of New Mexico
897	0101C3494	ARF

Table 21 (cont)

Code	Manufacturer's Designation	Manufacturer
898	Atpet 100	Atlas Powder Company
899	0301C3660	ARF
900	0401C3660	ARF
901	1001C3660	ARF
902	1101C3660	ARF
903	1102C3660	ARF
904	1103C3660	ARF
905	2201C3660	ARF
906	2301C3660	ARF
907	2401C3660	ARF
908	2402C3660	ARF
909	2501C3660	ARF
910	2502C3660	ARF
911	2601C3660	ARF
912	2602C3660	ARF
914	2702C3660	ARF
922	1101C3494	ARF
923	1102C3494	ARF

Table 22

COMPOSITION OF ARF FORMULATIONS

Code No.	Composition: Components	%	Blending Procedure
899	Barium dialkyl naphthalene sulfonate (351) 2,5-Dimethylpiperazine (477) *Phenothiazine (Thiodiphenylamine)	4.7 0.9 0.5	Components weighed and dissolved in carbon-treated Plexol 201 by heating on water bath until solution was effected (1-1/2 - 2 hr).
900	Barium dialkyl naphthalene sulfonate (351) 2,5-Dimethylpiperazine (477) Phenothiazine	5.0 1.0 0.2	Same as 899.
901	Barium dialkyl naphthalene sulfonate (351) 2,5-Dimethylpiperazine (477) Phenothiazine	5.0 2.0 0.2	A 0.4% stock solution of phenothiazine was warmed with the piperazine until solution was effected. This was diluted with a 10% stock solution of 351.
902	Barium dialkyl naphthalene sulfonate Phenothiazine	5.0 0.2	Equal volumes of the 10% and 0.4% stock solutions were mixed.
903	Barium dialkyl naphthalene sulfonate (351) Amine 220 (269) Phenothiazine	5.0 1.9 0.2	Amine 220 was dissolved in the 0.4% phenothiazine stock solution. This was mixed with an equal volume of the 10% 351 solution.
904	Barium dialkyl naphthalene sulfonate (351) 3,3'-Iminobispropylamine (471) Phenothiazine	5.0 1.9 0.2	Same as 903.
-	Due to the blending procedure, all formulations henceforth will contain 5.0% Barium dialkyl naphthalene sulfonate and 0.2% phenothiazine. Therefore, only the third component and its amount will be listed.		

Table 22 (cont)

Code No.	Third Component	%	Blending Procedure
905	3,3'-Iminobispropylamine salt of mahogany sulfonic acid (818)	2.0	A "combined stock solution" (8.333% of No. 351 and 0.333% of phenothiazine) was mixed with a 5% solution of the 3rd component in the proportion of 3:2 by volume.
906	m-Aminobenzotrifluoride salt of pelargonic acid** (694)	2.4	Same as 905
907	N-Aminopropylmorpholine salt of linoleic acid** (651)	2.0	Same as 905.
908	N-Aminoethylmorpholine salt of oleic acid (585)	2.0	Same as 905.
909	α-Methylbenzylamine salt of oleic acid (596)	2.0	Same as 905.
910	α-Methylbenzylmonoethanolamine salt of oleic acid (600)	2.0	Same as 905.
911	Morpholine salt of oleic acid (583)	2.0	Same as 905.
912	Tetraethylenepentamine salt of linoleic acid (645)	2.0	Same as 905.
914	3,3'-Iminobispropylamine salt of linoleic acid**(649)	2.0	Same as 905, except that hot solution of the salt was mixed with "combined stock solution" at room temperature to prevent precipitation.

*Crude material purchased from Eastman Kodak and recrystallized.

**Heated for 3 hr at 150°C.